PATENT SPECIFICATION

1,000,044

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Int. Cl.:—**G 08 c.**

COMPLETE SPECIFICATION

DRAWINGS ATTACHED

Improvements in or relating to the Testing of Metals

We, HOESCH AKTIENGESELLSCHAFT of 12, Eberhardstrasse, Dortmund, Germany, a German Company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:

The present invention relates to a method for determining chemical elements in metal articles or in melts, based on the principle of spectrum analysis of γ-radiation released by neutron irradiation.

In order to determine the elements present in metals, use has hitherto been made of chemical analysis methods, photo-optical analysis, Röntgen fluorescence analysis, mass spectrum analysis and activation analysis.

Observation by the latter analysis methods can only be effected in practice on quite small regions of the test material. This is quite unsatisfactory insofar as basically no homogeneous material exists. With activation analysis hitherto, the test sample was irradiated in a reactor with slow, so-called thermal neutrons, making radioactive the separate elements contained therein. The activated elements then emit an electromagnetic wave radiation and/or corpuscular radiation according to characteristic decay laws, and the radiations are then measured and evaluated.

This last method, despite its great advantages over previous methods, still has many disadvantages. Hitherto the test pieces generally had to be irradiated in a reactor, since the usual laboratory safe neutron sources which can be derived from e.g. a Po-Be reaction, have in general too small a neutron flux density. On the other hand, certain elements which are very important in steel analysis, especially oxygen, are only slightly activated by slow neutrons. Finally, the irradiated test piece, namely the metal body, still remains radioactive after testing with slow neutron irradiation.

However metal bodies have also been irradiated with slow neutrons using as source a deuterium-tritium reaction of an accelerator. By this d-t-reaction, fast neutrons in the MeV energy range are produced and these are transformed into slow neutrons, e.g. by a decelerating layer of paraffin. Basically the same disadvantages appear with this method as with reactor radiation.

Furthermore it is also well known, from mineral oil mining, how to determine the presence of certain elements in the interior of the earth by means of prompt γ-radiation which is released by fast neutrons and subsequently measured and evaluated.

According to the present invention, a method is provided for the determination of chemical elements in metal bodies or melts, in which at least part of the metal body or melt is bombarded with fast neutrons and the prompt γ-radiation released by the neutrons is measured and evaluated.

This new method has several advantages over the known methods of analysis based on the principle of spectrum analysis of radioactive radiation released by slow or decelerated neutrons. Above all, it makes possible the detection of oxygen, which is very important in metallurgy. In addition, this method produces hardly any activation of the test material and consequently hardly any radioactivity. Furthermore, because of the greater depth of penetration of the fast neutrons in combination with the penetrability of the prompt γ-radiation produced thereby, it

is also possible according to this method to analyse test pieces of greater thickness. Finally this method facilitates the determination of elements in the melt itself, which has hitherto been impossible.

In order to be able to determine, in particular, the existence of oxygen, the use of a d-t-reaction is recommended to produce fast neutrons, since of all the known fast neutron producing reactions only the d-t-reaction produces neutrons with sufficient energy for releasing prompt γ-radiation from oxygen. Naturally, for other elements, a different nuclear reaction can be used by which such fast neutrons are produced that the characteristic γ-radiation of the element under analysis is stimulated.

In order to identify and/or estimate the quantity of a single element, it is necessary to determine the energy distribution of the γ-radiation produced, so that measurement is thus effected according to energy levels. Measurement of the γ-radiation may also be made according to emission groups, however; for example, it may be desirable to determine the total non-metallic contents of an alloy and, as each of the respective elements emits γ-radiation of characteristic energy levels, these characteristic levels can be measured together in what is known as an "emission group." The measurement is carried out, for example, by a pulse height analyser coupled to the outlet of a radiation detector.

On analysing certain regions of the test material, it may be necessary to filter out the γ-radiation from other regions, in particular, the γ-radiation coming from the measuring system or from the media between the sample and the detector or from other regions of the text material. Preferably this can be carried out with collimators and/or masking systems mounted on one or more radiation detectors. However, it is also possible to arrange a neutron source and two or more radiation detectors in a tandem or telescopic arrangement connected with a coincidence circuit, so that only the γ-radiation which is incident simultaneously on the different radiation detectors is measured. The spatial cross-section of the assumed cone of neutron emission and γ-ray reception therefore determines the region of the test material being analysed.

In analysing large surfaces, it is preferable to start with a neutron source which has a cone of emission irradiating the entire test surface. The radiation detector may have a cone of reception including the entire test surfaces, but it may also have a smaller cone of reception and preferably may be guided continuously e.g. in raster form, over the test surface. Also the reverse arrangement of a scanning neutron source with a narrow cone of emission and a radiation detector with a fixed cone of reception including the entire test surface is possible. Furthermore, it is possible to allow a neutron source-radiation detector system with a small measuring range to scan a large test surface, preferably continuously. Finally, it is also possible to arrange plurality of neutron sources and radiation detectors adjacent one another. If necessary, it is possible to vary the size of the cone of emission as well as the cone of reception, so that the cones can be adjusted to the actual size of the test surfaces.

In analysing melts, e.g. in metallurgical furnaces or in casting ladles, it can be interesting to ascertain the proportion of certain elements in the metal bath as well as in the slag. This can be effected by adjustment of the elevation of the measuring system or by its pivotation about a horizontal axis, so that the point of measurement shifts from the metal bath to the slag and vice versa. It is also possible to make either the neutron source or the radiation detector movable and to make immovable the other part of the measuring system having a correspondingly large cone of emission or cone of reception. One possible form of indirect comparative measurement is if the neutron source, acting on the adjoining parts of the metal bath and slag, co-operates with one radiation detector directed on the metal bath and one directed on the slag zone.

The large depth of penetration of the fast neutrons as well as the penetrability of the prompt γ-radiation released thereby readily allows the measuring system to be set up outside a metallurgical furnace, such as a Siemens-Martin furnace.

The method according to the invention is suitable particularly for analysis during operation of the furnace and working of the metal, since results of the analysis can be continually and immediately read off after a first calibration.

Its use in practice is conceivable in all finishing steps in steel mills, beginning with the analysis of the melt, through the analysis of the intermediate product and up to the analysis of the finished product, and moreover during the finishing process itself. Basically the method can be carried out continuously or repeated at timed intervals.

The following is an example of an analysis for oxygen content:

A steel test piece of 100 grams in weight was bombarded with fast neutrons originating from a d-t-source. The pulse height analyser adjusted with its channels to measure γ-rays from 4.5 to 6.7 MeV energy measures the characteristics prompt γ-radiation occurring on interaction between these neutrons and the oxygen. The radiation detector counted 500 effective impulses in a period of for example 10 seconds, i.e. impulses free from foreign radiation. From a calibration curve determined by compara-

tive analyses or comparative measurements, it was seen that there was an oxygen content of 50 mg., corresponding to 0.05%.

It is clearly possible to determine an oxygen content even in the region of 0.001% with approximately 10% relative accuracy.

The method according to the invention can moreover be used advantageously for the non-destructive testing of metal with non-metallic inclusions or for locating shrinkage holes. As opposed to known methods for the non-destructive testing of metal bodies, as e.g. the ultrasonic testing method and the testing methods based on the radiation by Röntgen γ-radiation, the present method brings to light evidence of nonhomogeneity of the body and above all of its chemical state, on the basis of determining certain elements and if necessary their concentration.

According to the present method, it is readily possible for example to determine whether a shrink hole is clean or whether the inclusions connected with the welding contain e.g. oxide, sulphide or silicate.

The following is an example:

A hot unworked steel slab bloom rolled in an ingot slab bloom frame with the dimensions 105C mm. width, 150 mm. thickness and approx. 6000 mm. length, is bombarded with fast neutrons in a measuring unit before its head and tail are cut clean. The device continuously determines the amount of oxygen, sulphur and silicon therein. To do this for example, a measuring region of 100 mm. thickness, 20 mm. width and 20 mm. length is masked and this measuring region scans approx. 70% of the slab bloom relative to its width.

A multi-channel pulse height analyser gives the count for oxygen, sulphur and silicon in the tail of the slab bloom which can be compared with corresponding calibration curves showing the normal percentages of these elements in steel. These count values vary on scanning the slab bloom only immaterially until the measuring region scans a zone at approx. 1000 mm. distant from the head end of the slab bloom, the oxygen radiation intensity of which zone rises irregularly to approx. three times the amount of the normal value. This zone widens towards the head end of the slab bloom, and the intensity of oxygen radiation from the zone varies between three and ten times the value of the normal intensity. The same holds for the increase in the sulphur and silicon values.

The excess of oxygen, sulphur and/or silicon is evidence of the existence of a shrink hole at the head filled with oxides, sulphides and/or silicates, which hole because of the slag inclusions will not weld together with further rolling and preferably should be cut off as soon as possible.

In order that the invention may be more readily understood, the invention is described below in conjunction with the accompanying drawings, in which:

Fig. 1 shows schematically a test piece being irradiated by a radiation source and a radiation detector directed on the test piece;

Fig. 2 shows an arrangement similar to Fig. 1 with a pulse height analyser coupled to the output of the radiation detector;

Fig. 3 shows an arrangement similar to Fig. 1 with the radiation detector provided with a collimator attachment;

Fig. 4 shows an arrangement similar to Fig. 1 with several radiation detectors in a coincidence circuit;

Fig. 5 shows an arrangement similar to Fig. 1 with a radiation source and a radiation detector whose cone of emission and cone of reception respectively correspond to the size of the test piece;

Fig. 6 shows an arrangement similar to Fig. 1 with a radiation source whose cone of emission corresponds to the size of the test piece and a radiation detector movable over the test piece and having an area of reception for parallel radiation;

Fig. 7 shows an arrangement similar to Fig. 1 with a movable radiation source and a movable radiation detector;

Fig. 8 shows an arrangement similar to Fig. 1 with a plurality of radiation sources and radiation detectors;

Fig. 9 shows a cross-section through a metallurgical furnace with the melt which is being irradiated by a movable radiation source and on to which a movable radiation detector is directed;

Fig. 10 shows an arrangement similar to Fig. 9 with a radiation source simultaneously irradiating the metal bath and the slag and a movable radiation detector;

Fig. 11 shows an arrangement similar to Fig. 9 with a radiation source simultaneously irradiating the metal bath and the slag and having a radiation detector directed on the metal bath and a radiation detector directed on the slag.

Figs. 1 - 8 show a test piece 11 and its analysis zone 12, the source of radiation 13, a radiation source 113 with a large cone of emission, a movable radiation source 213, the radiation detector 14, a radiation detector 114 with a large cone of reception, a movable radiation detector 214, a pulse height analyser 16, a collimator 17 and an indicating or registering apparatus 18.

Figs. 9-11 show also a metallurgical furnace 9, with the slag 111 and the metal bath 211.

The separate known components of the device are shown quite schematically, partly by their standard symbols. Only the effective cross-section of the cones of emission and reception of the radiation source and radiation detector respectively are shown.

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It is clearly possible to determine an oxygen content even in the region of 0.001% with approximately 10% relative accuracy.

The method according to the invention can moreover be used advantageously for the non-destructive testing of metal with non-metallic inclusions or for locating shrinkage holes. As opposed to known methods for the non-destructive testing of metal bodies, as e.g. the ultrasonic testing method and the testing methods based on the radiation by Röntgen γ-radiation, the present method brings to light evidence of nonhomogeneity of the body and above all of its chemical state, on the basis of determining certain elements and if necessary their concentration.

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In order that the invention may be more readily understood, the invention is described below in conjunction with the accompanying drawings, in which:

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The separate known components of the device are shown quite schematically, partly by their standard symbols. Only the effective cross-section of the cones of emission and reception of the radiation source and radiation detector respectively are shown.

WHAT WE CLAIM IS:—

1. A method for the determination of chemical elements in metal bodies or melts, in which at least part of the metal body or melt is bombarded with fast neutrons and the prompt γ-radiation released by the neutrons is measured and evaluated.

2. A method as claimed in claim 1, in which the fast neutrons are produced by d-t-reaction.

3. A method as claimed in claim 1 or 2, in which the prompt γ-radiation is measured according to energy levels.

4. A method as claimed in claim 1 or 2, in which the prompt γ-radiation is measured according to emission groups.

5. A method as claimed in any preceding claim, in which the regions of the test material other than those to be analysed and the prompt γ-radiation, originating from other media are masked out.

6. A method for the determination of chemical elements in metal bodies or melts based on the principle of spectrum analysis of γ-radiation released by neutron irradiation, as hereinbefore described with reference to Fig. 1, Fig. 2, Fig. 3, Fig. 4, Fig. 5, Fig. 6, Fig. 7, Fig. 8, Fig. 9, Fig. 10 or Fig. 11 of the accompanying drawings.

POLLAK, MERCER & TENCH,
Chartered Patent Agents,
Audrey House, Ely Place,
London, E.C.1.
Agents for the Applicants.

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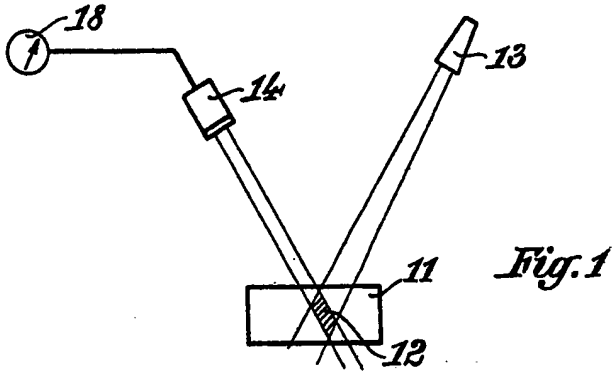
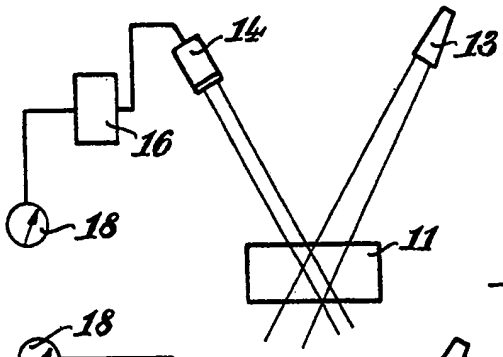


Fig. 1



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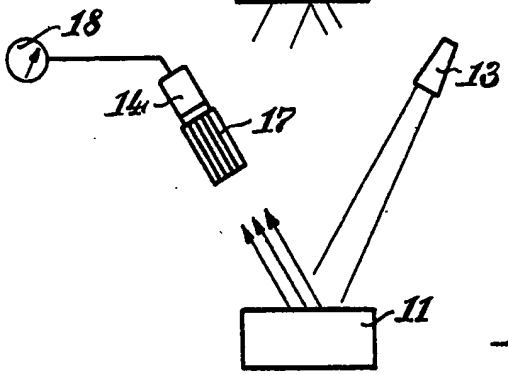

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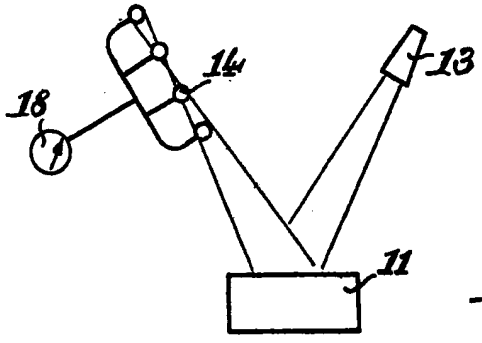

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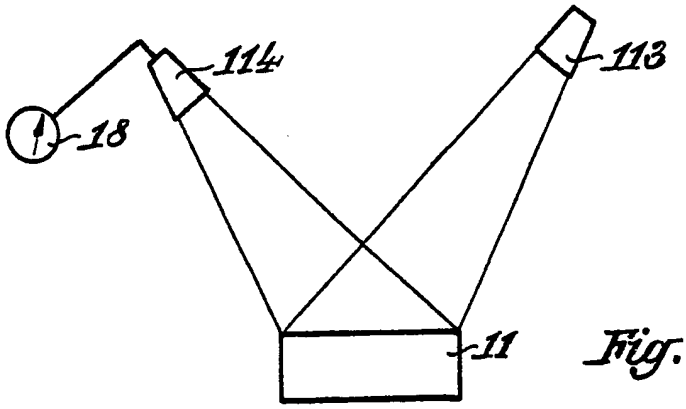

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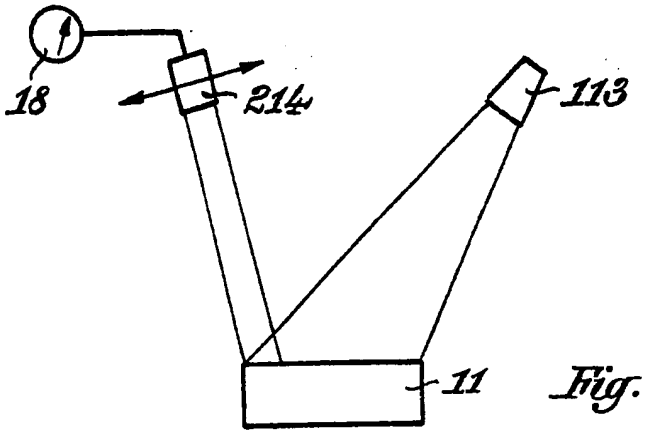

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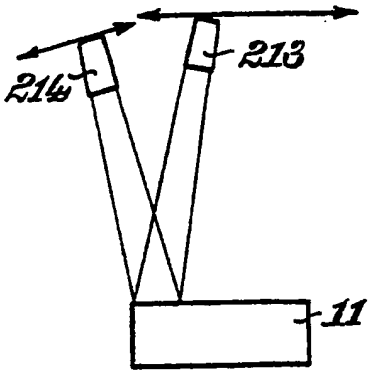

Fig. 7

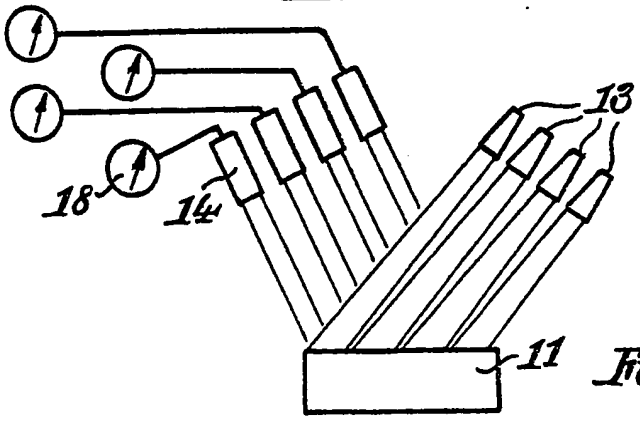

Fig. 8

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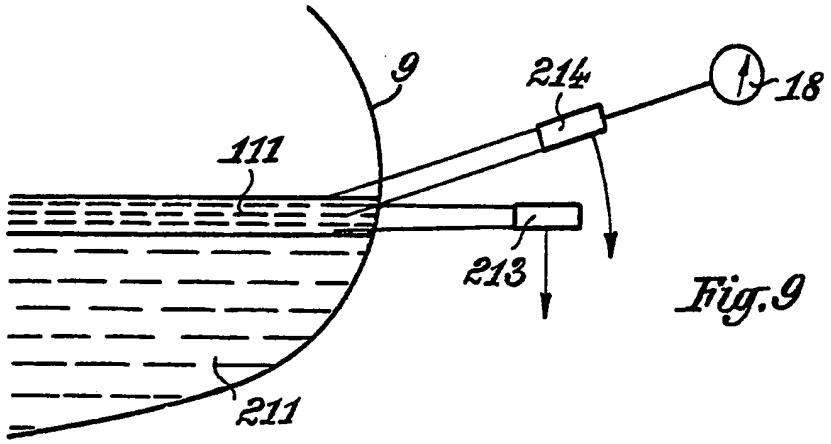

Fig.9

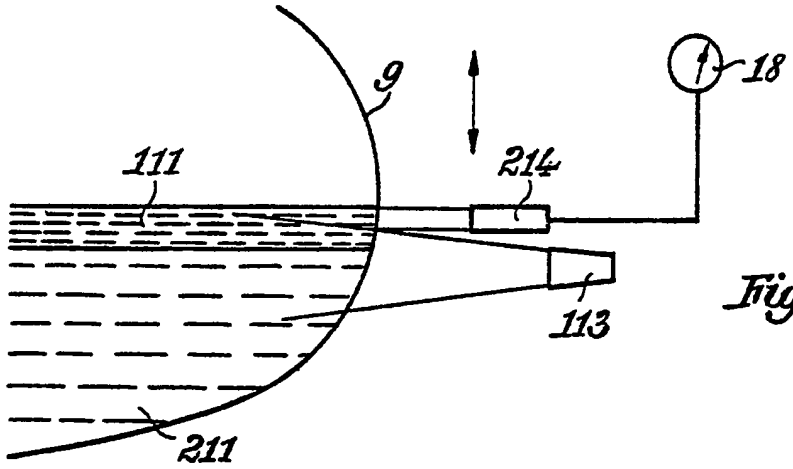

Fig.10

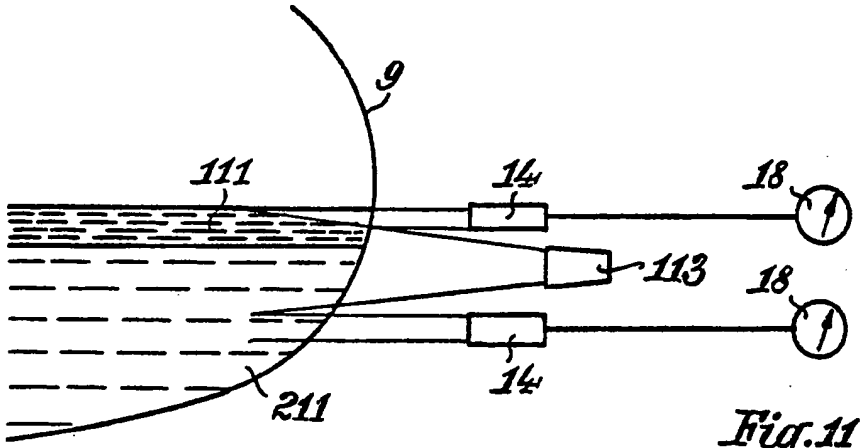

Fig.11

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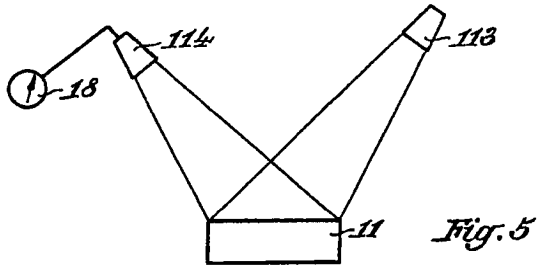

Fig. 5

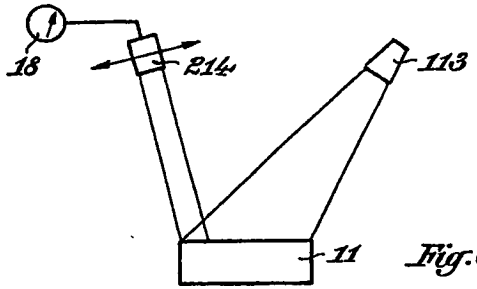

Fig. 6

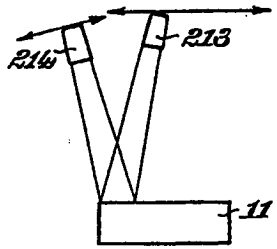

Fig. 7

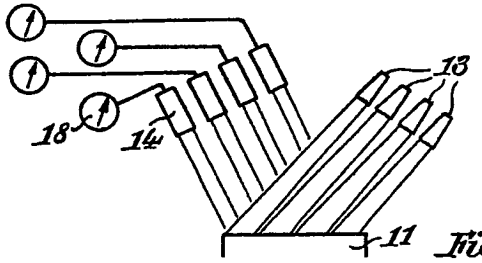

Fig. 8

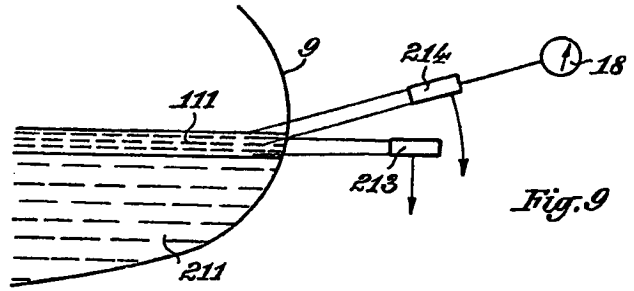

Fig. 9

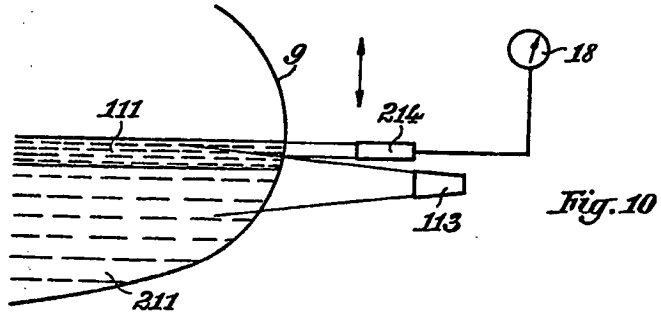

Fig. 10

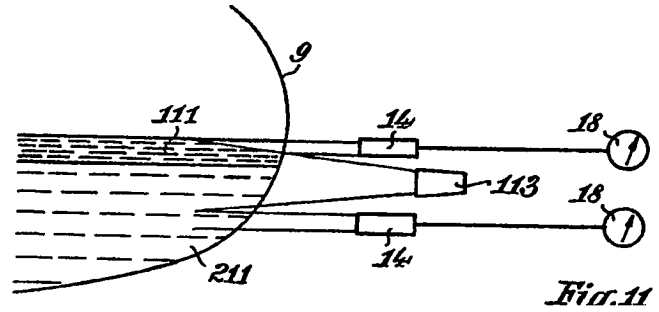

Fig. 11